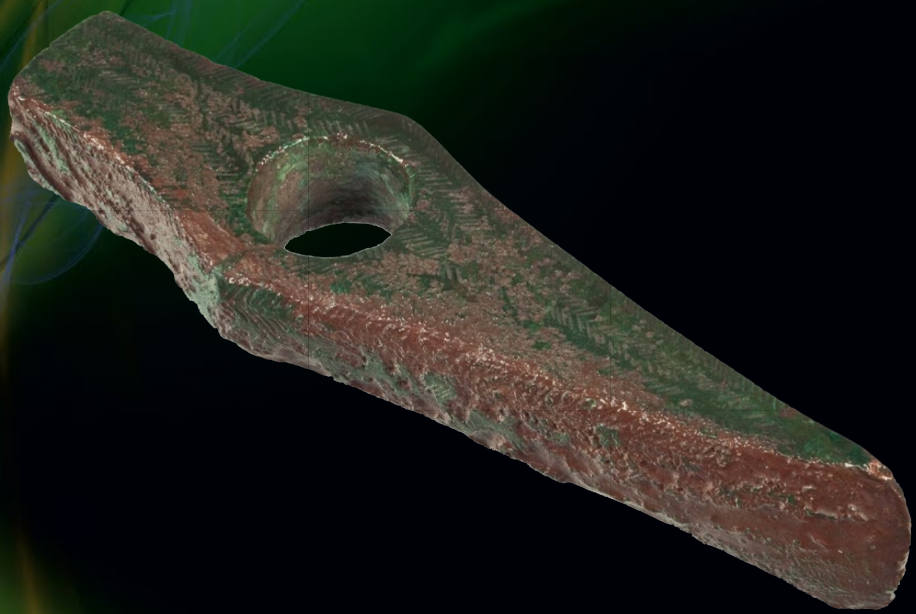
The Rise of Metallurgy in Eurasia

Evolution, Organisation and Consumption of Early Metal in the Balkans



Edited by

Miljana Radivojević, Benjamin W. Roberts, Miroslav Marić, Julka Kuzmanović Cvetković and Thilo Rehren

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Cover: Ljiljana Dinić; Copper hammer-axe, type Pločnik, c. 4600 BC
(from Pločnik, Serbia) - Julka Kuzmanović Cvetković.
Inner back cover: Reconstruction of the world's earliest copper smelting. Green flames come from the extraction of metal from malachite. Experiments at Pločnik, Serbia (2013) - Marko Djurica

Copyeditor: Kate Sharpe

To the memory of Borislav Jovanović, our colleague, friend and inspiration

(1930 – 2015)

Contents

Chapter 26

Pločnik: technology of metal production

Miljana Radivojević and Thilo Rehren

Metallurgical materials recovered during the excavation campaigns of 2012 and 2013 in Pločnik show similar characteristics to samples already studied and published previously (Radivojević 2012, 2015; Radivojević and Kuzmanović Cvetković 2014; Radivojević and Rehren 2016; Radivojević *et al.* 2013). They include, as for Belovode (Chapter 11), predominantly malachite minerals and ores (Table 1), that occur as roughly beneficiated pieces and without a distinct spatial patterning in Trench 24. In comparison to Belovode, they occur less frequently across all five horizons, partially explained by the fact that most of Trench 24 is a large rectangular feature – a house (F1=F2=F4=F5=F6=F10), and there is very little economic area surrounding it (Chapter 25, Figure 4).

The uncovered copper mineral samples are macroscopically similar to those already characteristic of both Pločnik and Belovode: dominated by green (malachite) minerals with black/dark specs (Figure 1b), and some that are more purely green, with an occasional occurrence of blue (azurite) (see Table 1). In addition to the expected malachite, Trench 24 produced an abundance of green-yellow minerals (Figure 1a) that reacted to the magnet used during the excavations (hence termed 'magnetic' in our archives). It is notable that these were found exclusively in Horizon 1 (see below). They were analysed along with the other metallurgy-related materials and the results are presented below.

Copper finds from Trench 24 can be separated into decorative minerals (malachite beads) and metal artefacts. The latter derive mostly from inside the large house in Horizon 1 (Chapter 25, Figure 4), but also include a copper metal ring (C_P2/13) found within Horizon 1, in Feature 3, external to the house (Figure 2).

A copper metal bead (C_P4/13) was found spatially associated with Feature 15, which is interpreted as a typical kiln in Horizon 3. Feature 3 is the only feature potentially related to metallurgical activities. It was detected adjacent to the west profile of the trench, close to corner A (Figure 2). This small feature consists of several large stones mixed with the remains of a burnt structure that could have been a kiln, or more likely a furnace, due to the find of a copper metal ring (Table 1) in its vicinity. The shape of Feature 3 is unusual (Figure 3): it appears to be a portion of a slightly elevated wall of an almost rectangular structure, or at least the edge of such a structure (see also Chapter 25, Figure 7). Its extent is unknown as it was not fully excavated (see Figure 3), however, it bears a striking resemblance to the excavated rectangular firing structures in Trenches 20 and 21 (Radivojević *et al.* 2013: 1033, Figure 2; Šljivar and Kuzmanović Cvetković 2009a: 61), which also had

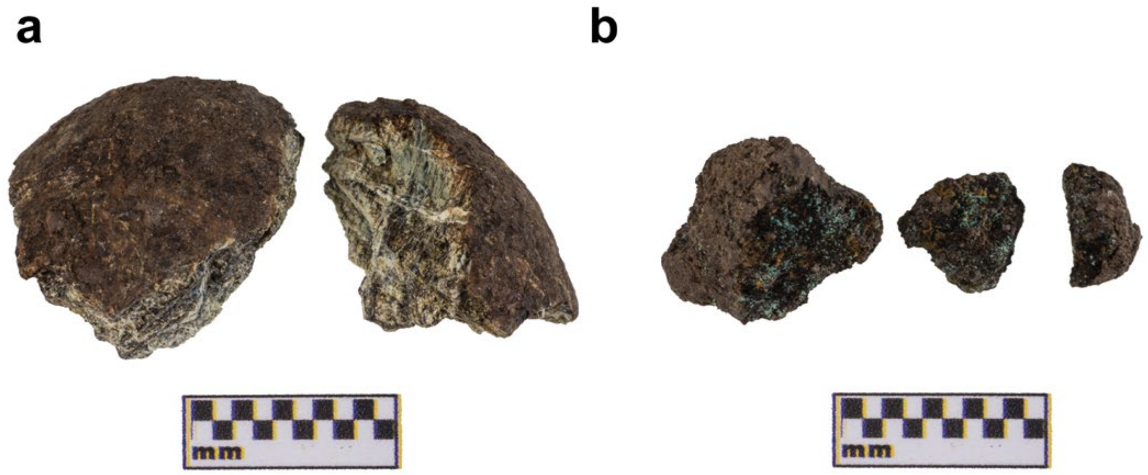

Figure 1. a) Example of green-yellow 'magnetic' mineral (P8/12); b) Example of black and green copper rich mineral (P125/13).

Table 1. The list of minerals and metallurgical materials from excavation campaigns 2012 and 2013 at Pločnik. Note an indicated subset analysed in depth with various analytical instruments.

No	no. finds	trench	spit	find no.	EDM	type of material	OM	metall-ogra-phy	SEM-EDS	EPMA	LIA	NAA	LA-ICP_MS	weight (g)	reasoning for provenance analysis
P6/12		24	S6	20	89	magnetic	X		X						
P14/12		24	S5	40	129	magnetic	X		X						
P58/12	1	24				bloated pottery	X								
P61/12		24	S8	96	283	metal droplet	X		X						
P117/12		24/F4	Sec 3	181	738	magnetic									
P8/13	2	T24	S10	214	977	magnetic					X	X			the earliest magnetic mineral
P10/13	2	T24	S10	220	988	fragmented metal	X	X	X	X		X	X	0.22	metal artefact
P13/13	1	T24	S11	233	1017	fragment of a bracelet/wire	X	X	X			X		0.15	metal artefact
P14/13	1	T24	S12	242	1054	malachite					X	X			malachite near P13/13
P18/13	1	T24	S14	272	1192	azurite									
P19/13	1	T24	S14	273	1197	malachite									
P55/13	1	T14/F15	S16	366	1606	malachite					X	X			ore choice consistency
P108/13	1	T24	S21	515	1904	malachite bead									
P117/13	2	T24	S21	530	1951	malachite bead x 2									
P121/13	1	T24	S21	537	1958	malachite					X	X			the last spit with minerals
P175/13	1	T24	S21	no	no	ceramic nozzle?									
P125/13	1	T24	S21	546	1967	malachite	X		X						

Table 1 continued. List of studied materials from Pločnik and analytical techniques applied

No	no. finds	trench	spit	find no.	EDM	type of material	OM	metall-ogra-phy	SEM-EDS	EPMA	LIA	NAA	LA-ICP_MS	weight (g)	reasoning for provenance analysis
C_P1/13	1	T24	S9	155	587	metal loop / ring	X	X		X	X	X	X	0.47	metal artefact
C_P2/13	1	T24/F2 (north)	S9	195	908	Metal band / ring	X	X		X	X		X	1.93	metal artefact
C_P3/13	1	T24	S10	217	985	Malachite bead									
C_P4/13	1	T24	S14	276	1200	Metal bead								0.05	
C_P5/13	1	T24	S18	405	1705	Malachite bead									
C_P6/13	1	T24	S21	516	1905	Malachite bead									
C_P7/13	1	T24	S21	524	1913	Malachite Bead									
C_P8/13	1	T24	S21	531	1952	Malachite bead									
C_P9/13	1	F32	S21	573	2078	Malachite bead									
C_P10/13	1	T24	S21	529	1950	Malachite bead flot									
C_P11/13	1	T24	S21	529	1950/2	Malachite bead flot									
C_P12/13	1	T24	S21	529	1950/3	Malachite bead flot									
Pf19/12		24 ext2		75	189	malachite bead									
Pf33/12		24	S8	96	283	magnetic	X		X					0.4	
Pf35/12		24	S8	102	297	magnetic	X		X					1.93	

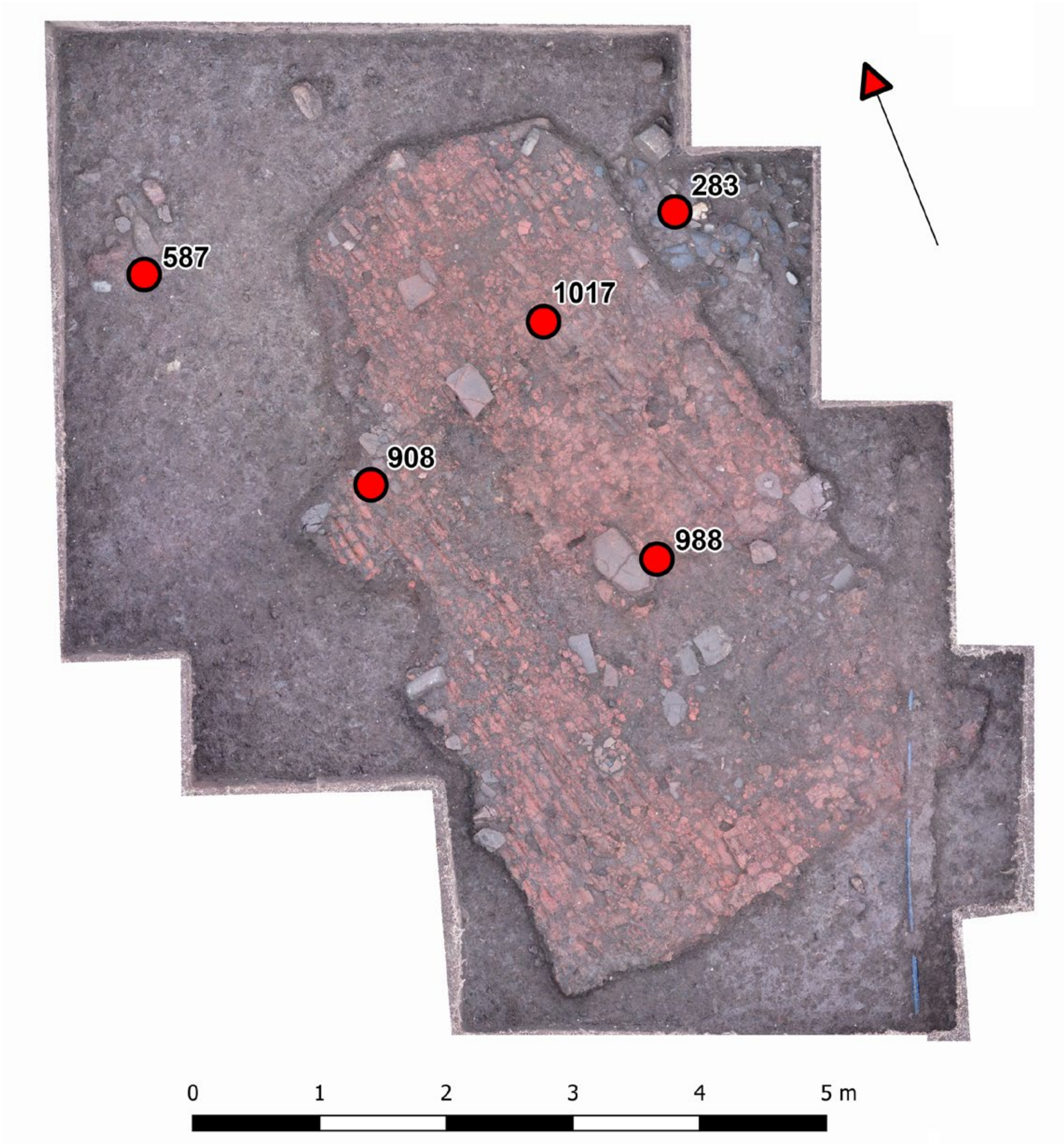

Figure 2. Overview of Horizon 1 house and spatial distribution of metal artefacts. All numbers are EDM identifiers for objects analysed in this chapter: P61/12 (283), C_P1/13 (587), C_P2/13 (908), P10/13 (988), P13/13 (1017) (prepared by M. Marić).

copper metal artefacts and casting debris associated with them. Feature 3 is distinct from the previous finds in that it was possibly located just outside a house but it would not be unusual to have the fireplace for metallurgical activities in an economic area, as seen at Belovode (Chapter 11). The excavated assemblage did not include any slag or slagged sherds, although the copper metal droplet (P61/12) may be indicative of what has previously been characterised as a 'slagless' metallurgy at Pločnik (Radivojević 2012; Radivojević and Rehren 2016: 220), with only one other previous find with a similar structure.

The direct radiocarbon dates for the house (F1=F2=F4=F5=F6=F10) and Features 3 and 15 frame the chronology of metallurgical finds from Trench 24 (see Table 2). In Chapter 37 (this volume), Marić *et al.* model the site chronology using the Bayesian statistical method, which combines both the radiocarbon dates and the relative stratigraphy recorded during the

Figure 3. Feature 3 in Trench 24. Note low rise red walls of what could have been a rectangular structure (centre).

excavation. These modelled dates are presented in Table 2, and we will refer to these when discussing the dating of metallurgical samples from Pločnik.

The modelled dates for Horizon 1 metallurgical samples (Table 2): P61/12, P10/13, P13/13, C_P1/13 and C_P2/13 indicate that they start in layers dated to 4631-4462 cal. BC to 4446-4231 cal. BC (95.4% prob.), or possibly 4576-4491 cal. BC to 4431-4324 cal. BC (68% prob.). This dating framework is also valid for all 'magnetic' minerals (see Table 1). The metal bead sample C_P4/13 belongs to Horizon 3, which starts at 5013-4968 cal. BC and ends at 4894-4747 cal. BC (95.4% prob.), or possibly 5036-4951 cal. BC to 4927-4621 cal. BC (68% prob.). This is consistent with the beginning of the Gradac Phase on the site (Chapters 25 and 37, Table 3), and is amongst the earliest securely dated copper metal objects from Pločnik, making it the earliest secure date for the beginning of metallurgy at the site.

Several fragments of a ceramic object from spit 21 (Horizon 4) could potentially resemble a tuyère (Figure 4), however a pXRF scan of the object did not show any contamination with potential ore elements either insides or outside, and the diameter of *c.* 2 cm is too large for the expected type of on-site activities. Interestingly, it was found in association with Feature 34, a large irregular rectangular area comprising fine white ash mixed with charcoal, daub fragments and baked soil. A radiocarbon date directly associated with this feature sets it at around 5003-4987 cal. BC (68% prob.), which is around the time that copper metallurgy emerges in Pločnik.

Methodology

The sampling and analytical strategy is identical to that applied at Belovode (see Chapter 11, Table 3). Polished blocks of copper metal artefacts C_P1/13, C_P2/13 and P10/13 were prepared for metallographic examination using, as an etchant, ammonia hydrogen peroxide made from equal proportions of ammonia (NH_4OH), water and 3% H_2O_2.

Results: technology of metal making and working in Pločnik

Ten samples were selected for in-depth microstructural and compositional analysis: five minerals/ores and five pieces of copper metal making and working evidence (a droplet and four fragments of copper metal artefacts). All provide evidence for copper-based metallurgy on the site which, in terms of the nature and volume of the process, is consistent with previous studies on archaeometallurgical materials both from this settlement and beyond, across the Vinča culture (Radivojević 2012, 2015; Radivojević and Rehren 2016).

Table 2. Direct dates for metallurgical activities in the site of Pločnik

No	trench	spit	find no.	EDM	Horizon	type of material	associated feature	modelled C14-date 1σ	modelled C14-date 2σ	directly dated feature/spit (2σ)
P61/12	24	S8	96	283	1	metal droplet	F1=F2=F4=F5=F6=F10	(start) 4576-4491 cal BC to (end) 4431-4324 cal BC	(start) 4631-4462 cal BC to (end) 4446-4231 cal BC	MAMS22083 4454-4356 cal BC
C_P1/13	T24	S9	155	587	1	Metal earing	F1=F2=F4=F5=F6=F10	(start) 4576-4491 cal BC to (end) 4431-4324 cal BC	(start) 4631-4462 cal BC to (end) 4446-4231 cal BC	MAMS22083 4454-4356 cal BC
C_P2/13	T24/F3	S9	195	908	1	Metal ring	F3	(start) 4576-4491 cal BC to (end) 4431-4324 cal BC	(start) 4631-4462 cal BC to (end) 4446-4231 cal BC	MAMS22081 4493-4365 cal BC
P10/13	T24	S10	220	988	1	metal foil corroded	F1=F2=F4=F5=F6=F10	(start) 4576-4491 cal BC to (end) 4431-4324 cal BC	(start) 4631-4462 cal BC to (end) 4446-4231 cal BC	MAMS22083 4454-4356 cal BC
P13/13	T24	S11	233	1017	1	metal bracelet (?)	F1=F2=F4=F5=F6=F10	(start) 4576-4491 cal BC to (end) 4431-4324 cal BC	(start) 4631-4462 cal BC to (end) 4446-4231 cal BC	MAMS22083 4454-4356 cal BC
C_P4/13	T24	S14	276	1200	3	Metal bead	F15	(start) 5013-4968 cal BC to (end) 4894-4747 cal BC	(start) 5036-4951 cal BC to (end) 4927-4621 cal BC	MAMS22090 4937-4796 cal BC

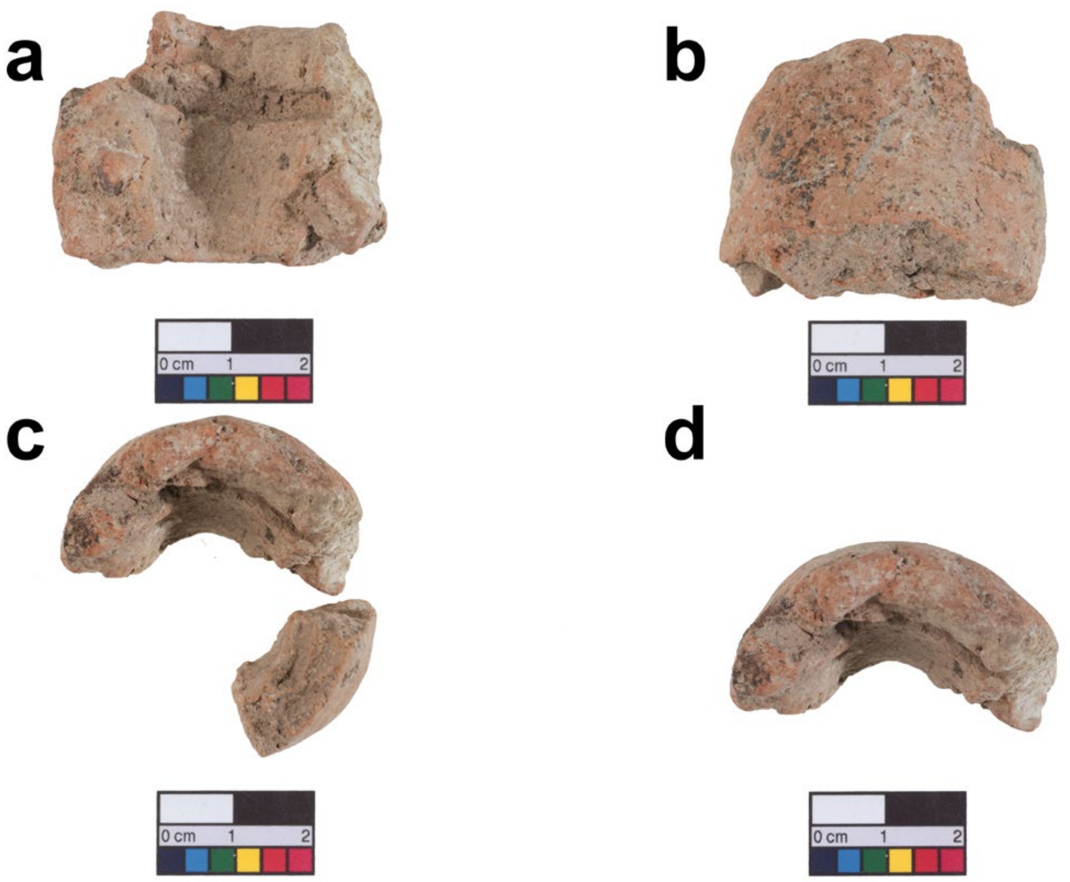

Figure 4. A cylindric ceramic artefact from Horizon 4, Pločnik.

Processing: archaeological minerals

Of the five minerals/ores analysed, one was malachite (P125/13, see Figure 1b, Table 3) and four belonged to the category of 'magnetic' minerals (Figure 6), that were more unusual and clearly different from the typical black and green copper-rich minerals that regularly occur at both Pločnik and Belovode. The black and green copper mineral, P125/13, contains both copper and manganese phases, with Mn content in the latter close to 50 wt% (Table 3). The so-called 'magnetic' minerals, P6/12, P14/12, Pf33/12 and Pf35/12 are a mixture of iron oxides and members of the olivine family of minerals (Table 4). The lightly coloured phase (pale yellow) is iron oxide, and the darker phase (see Figure 6) includes forsterite (Mg-end member of the olivines) and monticellite ($CaMgSiO_4$). In all cases, the texture of the samples identified them as natural rock fragments rather than metallurgical products. Their distinctive colour (mostly dark green) may explain their collection by the Pločnik communities. We do not currently know whether they were somehow related to the smelting process, since our evidence is for 'slagless' smelting processes (see below), or whether they are unrelated to metallurgy. Their presence does, however, seem to be anthropogenic, since such finds are not known from other excavations in Pločnik.

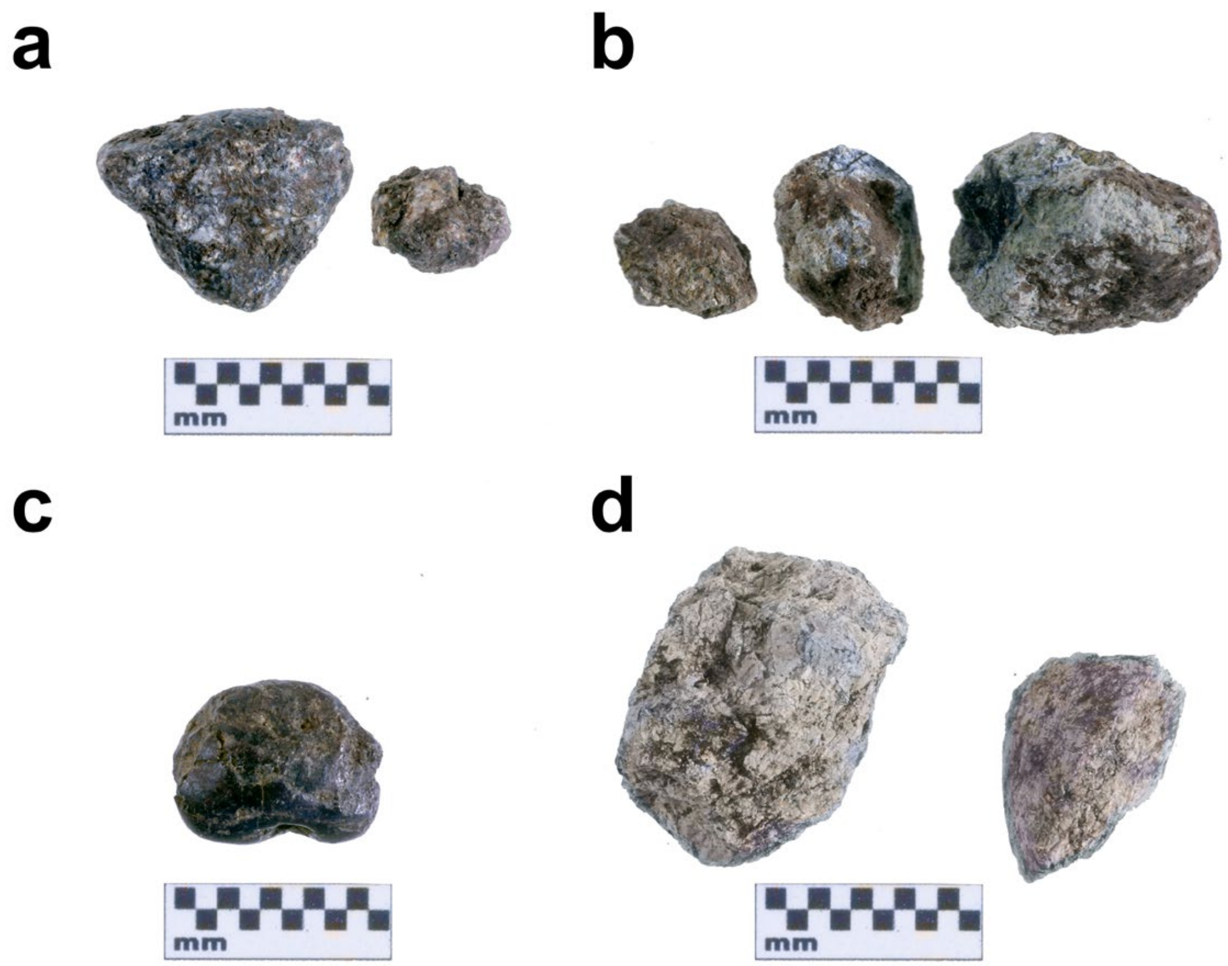

Figure 5. 'Magnetic' minerals. a) P6/12; b) P14/12; c) Pf33/12; d) Pf35/12.

Table 3. SEM-EDS compositional data for copper (green) and manganese (black) rich phases in P125/13. All values are averages of six to fourteen analyses of each sample / phase and normalised to 100%.

	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	MnO	FeO	NiO	CuO	ZnO	PbO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
P125/13 green	0.0	0.0	0.0	0.0	0.0	0.0	0.7	0.8	0.0	97.2	2.2	0.0
P125/13 black	0.0	2.4	6.1	0.9	0.0	1.0	47.6	7.2	0.0	12.8	4.0	18.4

Table 4. SEM-EDS compositional data for 'magnetic' minerals, with iron oxide (light) and olivine (dark) rich phases. All values are presented as stoichiometrically calculated at% and averages of two to nineteen analyses of each sample / phase and corrected with factors based on CRM analysis; the uncorrected data is reported in the Appendix B_Ch26.

	Mg	Al	Si	P	Ca	V	Cr	Mn	Fe	Ni	O
	at%	at%	at%	at%	at%	at%	at%	at%	at%	at%	at%
P6/12 light	0.0	0.0	0.0	0.0	0.0	0.2	0.0	0.0	49.7	0.0	50.1
P6/12 dark	11.9	9.9	12.0	0.0	0.1	0.0	0.0	0.1	8.7	0.0	58.5
P14/12 light	9.7	1.8	19.0	0.0	9.6	0.0	0.3	0.0	0.7	0.0	60.0
P14/12 dark	22.4	1.0	17.0	0.0	0.2	0.0	0.0	0.0	3.2	0.0	58.7
Pf33/12 light	0.0	0.0	2.6	0.0	0.1	0.0	0.0	1.0	44.9	0.0	51.3
Pf35/12 light	1.4	0.0	1.4	0.0	0.0	0.0	0.0	0.0	46.6	0.1	50.7
Pf35/12 dark	24.1	0.4	16.8	0.0	0.1	0.0	0.0	0.0	2.6	0.0	58.5

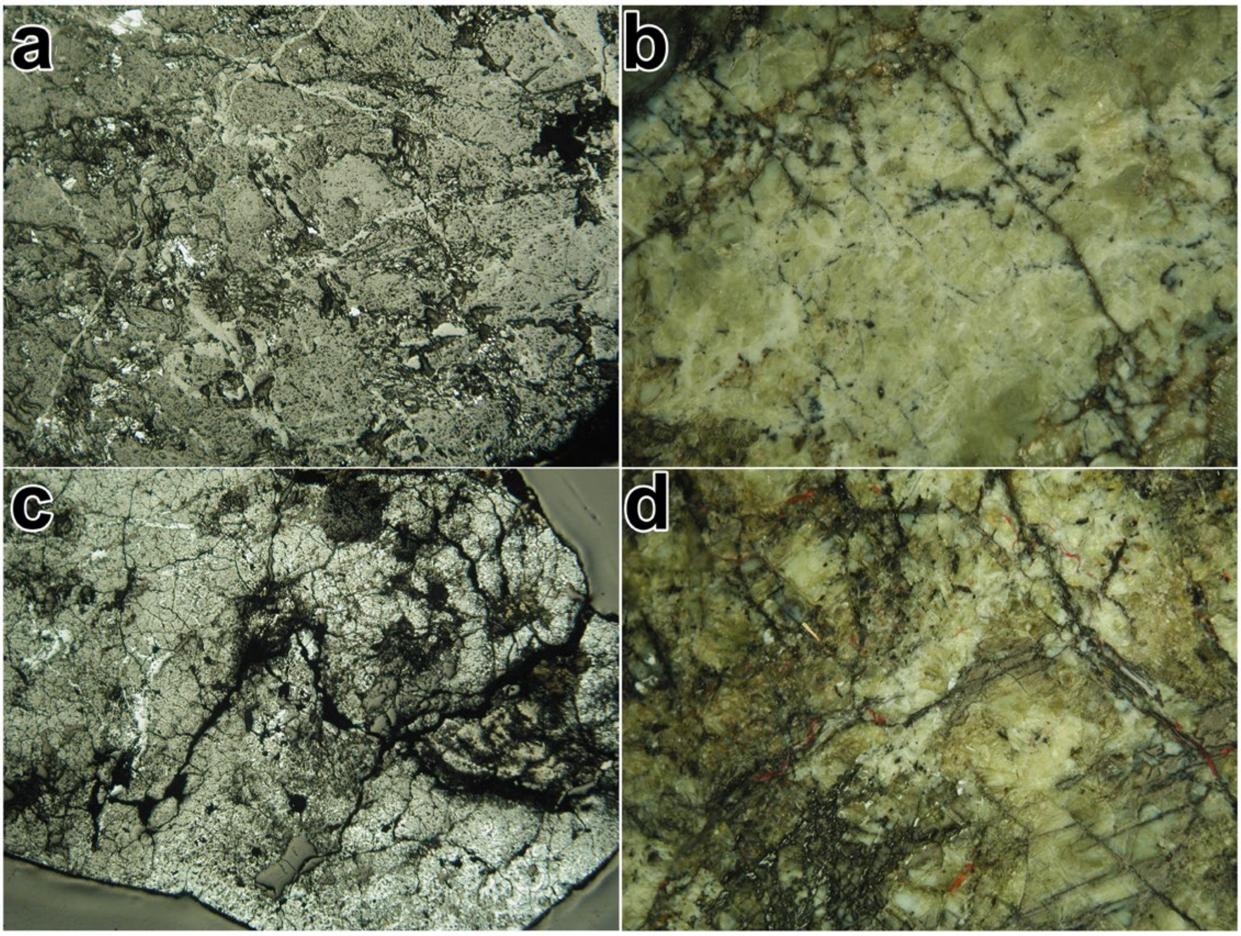

Figure 6. a) Photomicrograph of P6/12 taken under plain polarised light, 25x magnification, 6.4 mm width. Note that the lighter phase is iron oxide, while darker belongs to olivine family; b) photomicrograph of P14/12 taken under cross polarised light, 50x magnification, 3.2 mm width; c) photomicrograph of Pf33/12 taken under plain polarised light, 25x magnification, 6.4 mm width; d) photomicrograph of Pf35/12 taken under cross polarised light, 50x magnification, 3.2 mm width.

Making and working: copper minerals and metal artefacts

The total assemblage of copper-related artefacts from Pločnik includes thirteen mineral ornaments, one copper metal droplet, and fragments of five finished copper metal artefacts. Almost all are produced from copper-rich minerals or directly linked to metallurgy, demonstrating local metal smelting and working activities.

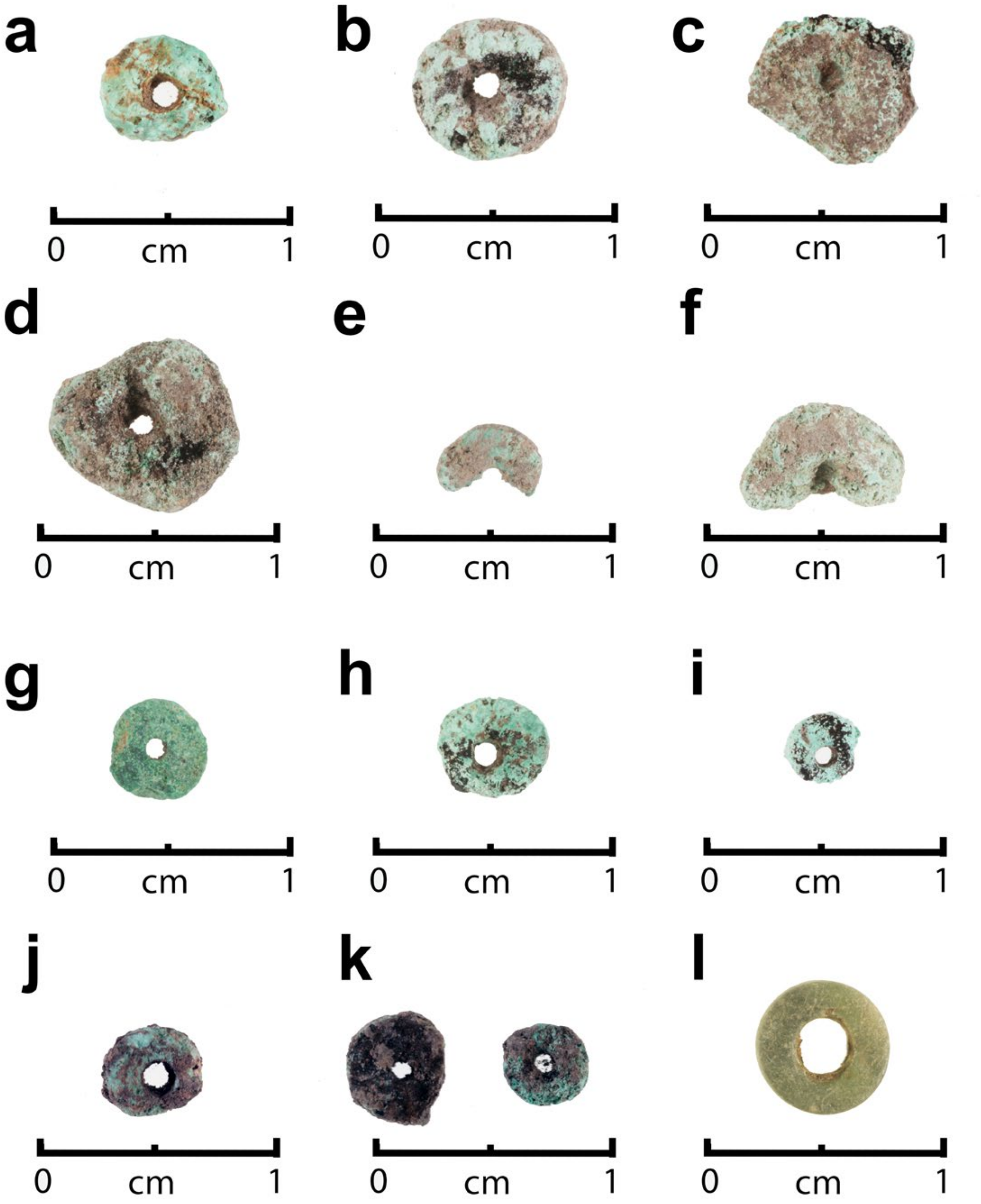

Figure 7. Mineral beads from Trench 24, Pločnik. a) C_P3/13; b) C_P5/13; c) C_P6/13l; d) C_P7/13; e) C_P8/13; f) C_P9/13; g) C_P10/13; h) C_P11/13; i) C_P12/13; j) P108/13; k) P117/13; l) Pf19/12.

Copper mineral artefacts (ornaments)

Thirteen mineral ornaments (beads) were found, of which all but one (Pf19/12, green stone ring bead, Figure 7/l) are made of malachite of varying purity (Figure 7)., The malachite beads can be roughly divided into three distinctive typological categories: circular or cylindrical (C_P3/13, C_P5/13, C_P7/13, C_P8/13, C_P9/13, C_P11/13, P108/13, P117/13); flat disc (C_P6/13, C_P10/13); and ring beads (C_P12/13) (cf. Wright *et al.* 2008; Wright and Garrard 2003). Visually, all beads show a thick, light green corrosion layer, while seven of the thirteen (C_P5/13, C_P7/13, C_P9/13, C_P11/13, P117/13; Figure 7b, 7d, 7f, 7h, 7k) have visible black (likely manganese-rich) areas. All but one are finished objects, fragmented after use or during the post-depositional processes. The exception is C_P6/13 (Figure 7c), which is a blank with an unfinished central hole indicating local bead manufacture.

Copper metal droplet

As noted in Chapter 11 on Belovode metallurgy, the 'Droplet' category of metallurgical debris can include semi-molten and fully molten pieces of ore/metal that could be produced by a wide range of activities. These droplets can represent anything from an attempt to smelt metal through to accidental loss of debris from smelting, melting or casting; most importantly, they are not worked any further, and this distinguishes them from the category of copper 'Artefacts'. One object, P61/12, recovered from Trench 24 in 2012, fits

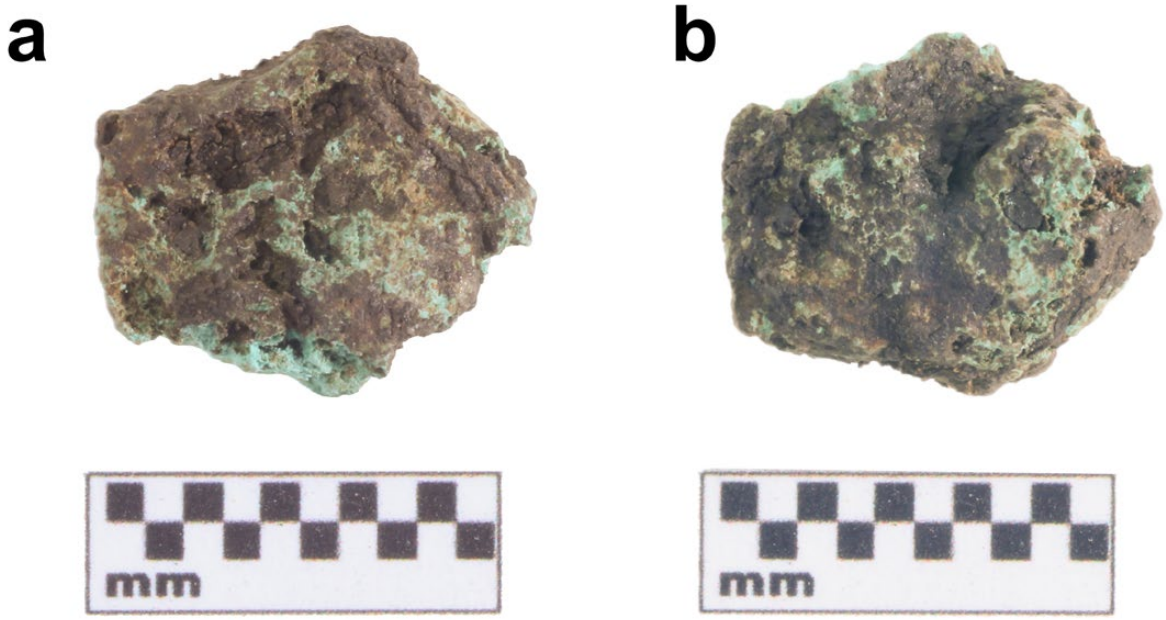

Figure 8. Metal droplet P61/12. Note green staining on top.

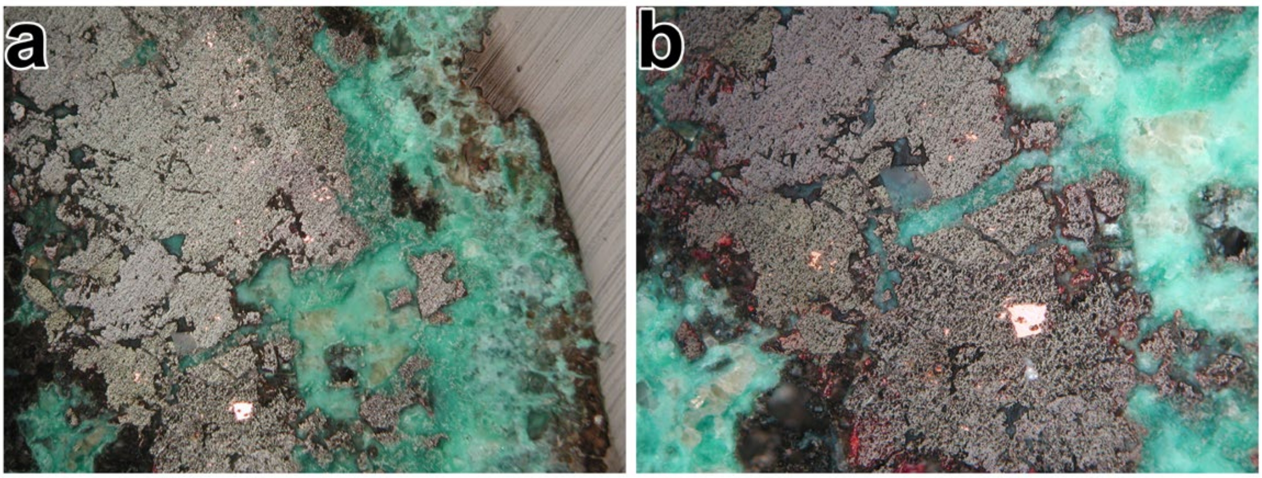

Figure 9. a) Photomicrograph of P61/12 taken under cross polarised light, 50x magnification, 3.2 mm width. Note bright yellow residual copper metal in a matrix of copper oxide (grey) and green corrosion products; b) photomicrograph of P61/12 taken under cross polarised light, 100x magnification, 1.6mm width.

Table 5. SEM-EDS compositional data for metal droplet P61/12, both metal and dross phase. All values are averages of four to fifteen analyses of each sample / phase.

	O	S	Cu	Zn
	at%	at%	at%	at%
P61/12 metal	1.7	0.0	98.3	0.0
stdev.s	*0.3*	*0.0*	*0.3*	*0.0*
P61/12 dross	33.6	0.7	65.7	0.0
stdev.s	*2.7*	*2.8*	*2.9*	*0.0*

the 'Droplet' criteria (Figure 8). It was found within the house in Horizon 1 and is not associated with any firing feature. The droplet has an amorphous shape, is covered in thick green patina, and is less than 1 cm in length. As with previous, similar, examples from Belovode (Chapter 11; see also Radivojević and Rehren 2016), this sample was initially collected and labelled as a mineral, its partially metallic structure only being revealed when it was cut with a saw (Figure 9). The droplet contains predominantly dross (copper oxides) with small specs of bright yellow copper metal that indicates what must initially have been a fully developed metallic phase. Optically, the dross is characterised by bright red internal reflections under cross polarised light, typical for cuprite (Cu_2O) (Figure 9). The presence of sulfur (Table 5) implies that the ore used contained both primary and secondary copper mineralisations. Overall, P61/12 can be interpreted as deriving from a 'slagless' melting event as there is no iron detected (see Table 5), or from a smelting event using copper ore of high purity. Experiments with copper smelting and refining have yielded similar material, retained on the crucible wall due to insufficiently high temperatures (i.e. only just above the melting point), or the presence of impurities (S. Timberlake, personal communication). Since it was found inside the dwelling, we assume that this droplet was in a secondary location, away from the location of high temperature processing where it was produced.

Copper metal fragments

Four of the five copper metal artefacts were analysed in detail with the results presented here, while the fifth was used entirely for provenance analysis (see Chapter 41). All four examined artefacts originate from Horizon 1 (Figure 2) and are associated with the house (F1=F2=F4=F5=F6=F10) and fireplace (Feature 3) in its vicinity (C_P2/13). Two artefacts appear to have been uncovered in their final form: an earring/loop (C_P1/13) and a (band) ring (C_P2/13); the other two are potentially fragments of unspecified decorative objects.

C_P1/13 is a loop or a ring, almost 1 cm in diameter (Figure 10a). It has a pale yellow, bright (copper) metal body, with green corrosion products developing on its edges (Figure 11). The object has been cast, and then worked with a combination of techniques carefully designed to respond to the desired function of the object. It has a fully recrystallised microstructure that was initially cold worked and then annealed, as evidenced by the presence of annealing twins (Figure 11b). The object must have been subject to several cycles of cold working and annealing, given the small size of the grains, the shape of the object, and also the noticeable folding line, implying that it began as a sheet of copper metal that was worked by folding onto itself. The final stage of production is preserved as deformed annealing twins, which commonly originate from low temperature mechanical or thermal stressing (Rostoker and Dvorak 1990: 23): the presence of twinned slip lines suggests that the final stage in the making of this artefact was cold working.

C_P2/13 also exhibits a cold worked microstructure, with the Cu+Cu_2O eutectic deformed into distinct layers (Figure 12). The fully recrystallised grain structure presents traces of several cycles of annealing (annealing twins) and cold working and distortion (slip lines, mechanical deformation, deformed annealing twins). These repeated cycles are further indicated by the reduction in grain size by *c.* 20% (Rostoker and Dvorak 1990: 16), however the reduction in thickness did not cause intolerable brittleness for this object. The final stage of manufacture of this piece of folded metal was also cold working.

Based on its shape, P10/13 was most likely once a decorative item, or potentially a piece preserved as metal stock (Figure 10d). The fully recrystallised grain structure developed within a regularly distributed copper–copper oxide eutectic (Figure 13). Annealing twins, slip lines, and deformed twin lines all indicate that this object was worked in the same manner as the rest of the assemblage: cold working followed by annealing, in several cycles. The microstructure seems fully recrystallised and the reduced grain size (from working) corroborates the evidence for cycles of annealing and cold working, or recrystallisation at temperatures low enough to prevent further grain growth (American Society for Metals 1979: 60). Judging by the elongation of grains and the separation of the copper–copper oxide eutectic into distinct layers, as well as the greater presence of slip lines, it appears that one side of the artefact was worked more intensively than the other (Figure 13a). The concentration of slip lines towards the working surface (Figure 13a) indicates that the final stage of manufacture was cold finishing, or they may have resulted from use of the finished object. The other side of the object preserves the dendritic structure of the initial cast, next to a significant amount of copper oxide inclusions, segregated towards the

Figure 10. Copper metal artefacts from Trench 24, Pločnik. a) C_P1/13; b) C_P2/13; c) C_P4/13; d) P10/13; e) P13/13.

surface. This side bears some traces of annealing twins (Figure 13b), suggesting that it was also hammered, although not as extensively as the other side. Of note are two distinct layers of corrosion in the structure of the artefact. These could have results from it being folded onto itself, either during production, or when in use.

A fragment of a bracelet or wire (P13/13) is a completely corroded form of what was once a copper metal body (Figure 14). Specks of bright yellow copper metal in the predominantly oxidised copper (dross) matrix confirm this. It is unclear what the object might originally have been, however judging by its shape it may have been a

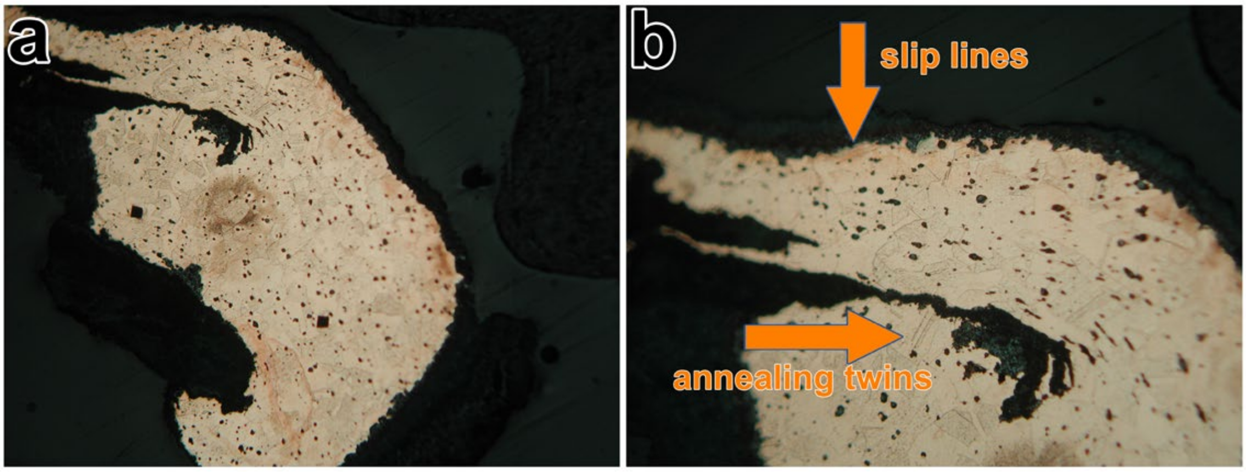

Figure 11. a) Photomicrograph of C_P1/13, taken under plain polarised light, 100x magnification, 1.6 mm width, etched with ammonia hydrogen peroxide; b) photomicrograph of C_P1/13, taken under plain polarised light, 200x magnification, 0.85 mm width, etched with ammonia hydrogen peroxide. Note the folding line in the middle.

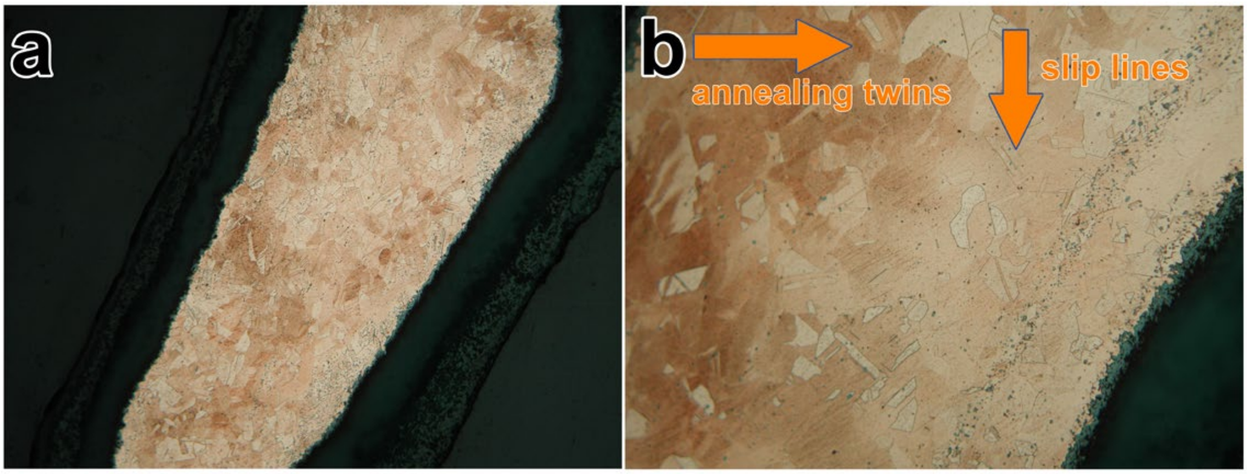

Figure 12. a) Photomicrograph of C_P2/13, taken under plain polarised light, 50x magnification, 3.2 mm width, etched with ammonia hydrogen peroxide; b) photomicrograph of C_P2/13, taken under plain polarised light, 200x magnification, 0.85 mm width, etched with ammonia hydrogen peroxide. Note annealing twins and slip lines.

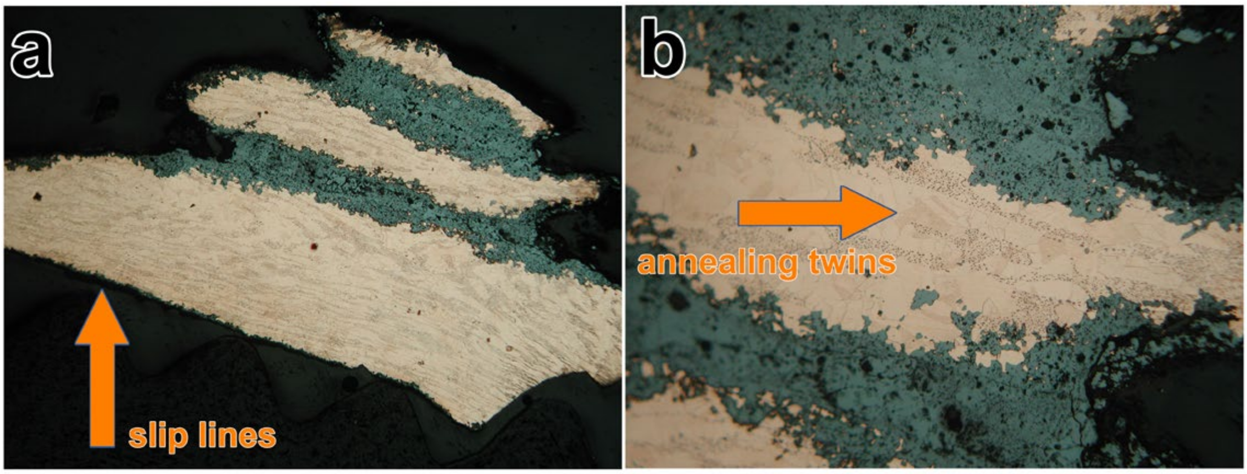

Figure 13. a) Photomicrograph of P10/13, taken under plain polarised light, 50x magnification, 3.2 mm width, etched with ammonia hydrogen peroxide. Note one side (bottom) worked harder than the other; b) photomicrograph of P10/13, taken under plain polarised light, 200x magnification, 0.85 mm width, etched with ammonia hydrogen peroxide.

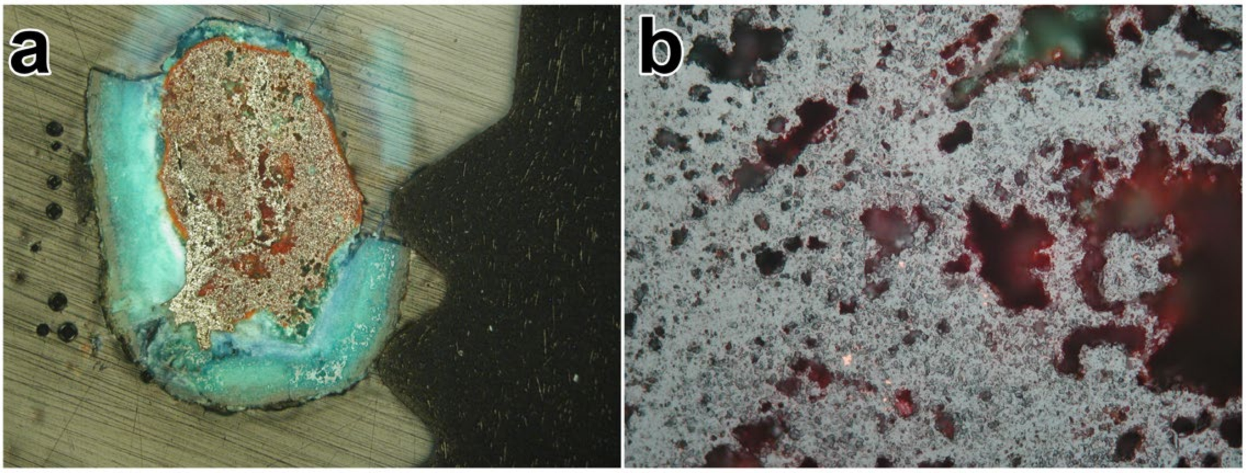

Figure 14. a) Photomicrograph of P13/13, taken under plain polarised light, 50x magnification, 3.2 mm width, etched with ammonia hydrogen peroxide; b) photomicrograph of P13/13, taken under plain polarised light, 500x magnification, 0.34 mm width, etched with ammonia hydrogen peroxide.

decorative. As such, it fits into the overall picture of an assemblage of exclusively decorative metal items from Trench 24.

While all the objects are made from copper of high purity, electron probe examination offers insight into the significantly different trace element signatures of three of them: P10/13, C_P1/13 and C_P2/13 (Table 6). We use ppm (parts per million) to discuss μg/g (microgram per gram) concentrations in this text. P10/13 is made of very pure copper with readings of Ni and possibly S; C_P1/13 is dominated by Pb (0.2 wt% on average) and Ag (*c.* 200 ppm on average) as main impurities; and C_P2/13 shows a significant reading of S (*c.* 160 ppm on average) and Sn (*c.* 100 ppm). The iron content is low in all three, suggesting either that the copper was highly purified (by multiple melting episodes), or that they all stem from a copper ore of high purity. The difference in trace element signatures would imply that each artefact derives from a different ore batch. This conclusion is strengthened with LA-ICP-MS results (Table 6a), with largely consistent results when it comes to the dominant Ni content in the copper ore used to make these artefacts. The Pb content, however, comes out as significant (c. 1.8 wt%) in C_P1/13. While it clearly shows lead content in the smelted copper ore, it is possible that the laser ablation picked up a phase rich in lead in the analysed metal section (copper and lead do not mix well).

Discussion and conclusion

The metallurgical evidence from Pločnik fits well within the broader picture developed in previous work and publications (Radivojević 2012, 2015; Radivojević and Kuzmanović Cvetković 2014; Radivojević and Rehren 2016; Radivojević *et al.* 2013). The copper metal droplets from previous research have already established the 'slagless' metallurgy principle at the settlement, and while evidence for primary production of copper may still be present, it remains to be found in future excavations. However, the new finds from Pločnik confirm it as a place for secondary processing (such as melting/refining), and we have been able to directly date a firing structure (Table 2) that, potentially, looks similar to several others discovered in its vicinity, and is associated with finished and semi-finished copper and tin bronze artefacts (Radivojević *et al.* 2013; Šljivar and Kuzmanović Cvetković 2009a).

That Pločnik belonged to what we have termed a multi-consumer metallurgical network is not novel information; Radivojević *et al.* (2010) previously established the link between metal production at Belovode and some artefacts from Pločnik. Prior to that, Pernicka *et al.* (1993; 1997) identified at least three different sources of copper that supplied this settlement from across the Balkans. In their application of the complex networks modularity method, Radivojević and Grujić (2018) were able to trace, in high resolution, the shifting nature of copper metal supply to Pločnik, which adjusted twice during the course of the Vinča culture: 1) when copper mineral use shifted to extractive metallurgy, in *c.* 5000 BC; and 2) when Vinča culture sites in the north were abandoned and new economic relationships forged with KGK VI cultural complex communities in Bulgaria (see also Figures 9-11 in Chapter 3, this volume). We can see, therefore, that the Pločnik communities were able to shift their subsistence economy towards the east of the Balkans, which may have contributed to extending the lives of these communities well into the 44th century BC (see Chapter 37, Table 3), almost 200 years longer than Belovode, and other key Vinča sites north from Pločnik. In Chapter 41 we will synthesise the metal technologies of both Belovode and Pločnik, and present a detailed exploration of new provenance data for both sites, feeding into a comprehensive

Table 6. EPMA compositional data of metal artefacts P10/13, C_P1/13 and C_P2/13 (selected significant trace element values), given in wt%. Values above *c.* 0.01 wt% (100 ppm) are considered reliable based on CRM measurements; values below this are indicative only. All data are corrected for values obtained from the reference material, using a procedure reported in the methodology section of Belovode metallurgy (Chapter 11). Values sought but not found at levels above *c.* 0.01 wt% were indicated as not detected (n.d.).

	S	Mn	Fe	Co	Ni	As	Ag	Sn	Te	Au	Pb	Bi	Analytical Total
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	
P10/13	0.005	0.001	0.002	n.d.	0.008	n.d.	n.d.	0.030	n.d.	n.d.	0.008	n.d.	99.93
P10/13	n.d.	0.003	0.001	n.d.	0.016	n.d.	n.d.	n.d.	0.014	n.d.	n.d.	n.d.	99.95
P10/13	0.002	0.005	n.d.	n.d.	0.011	n.d.	n.d.	n.d.	0.015	n.d.	n.d.	n.d.	99.93
P10/13	0.001	0.011	n.d.	0.005	0.012	0.051	n.d.	0.016	n.d.	0.023	n.d.	0.017	99.65
P10/13	0.001	n.d.	0.001	0.009	0.012	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	99.58
P10/13	0.001	n.d.	0.002	n.d.	0.019	0.005	n.d.	n.d.	0.004	n.d.	0.001	0.023	99.44
P10/13	0.005	0.001	n.d.	n.d.	0.015	n.d.	n.d.	0.002	0.003	n.d.	n.d.	n.d.	99.61
P10/13	0.004	n.d.	0.001	0.002	0.010	n.d.	0.011	n.d.	0.002	n.d.	n.d.	0.078	99.73
P10/13	0.005	0.005	n.d.	n.d.	n.d.	0.022	0.003	0.014	n.d.	n.d.	n.d.	0.017	99.59
P10/13	n.d.	n.d.	0.003	n.d.	0.004	n.d.	0.007	0.002	n.d.	n.d.	0.038	n.d.	99.77
C-P1/13	0.001	0.003	n.d.	n.d.	0.007	n.d.	0.024	n.d.	0.028	n.d.	0.280	n.d.	99.61
C-P1/13	n.d.	n.d.	0.002	0.004	0.006	n.d.	0.007	0.005	n.d.	n.d.	0.419	n.d.	99.34
C-P1/13	0.001	n.d.	0.008	n.d.	0.005	n.d.	0.019	0.006	n.d.	n.d.	0.260	n.d.	99.62
C-P1/13	0.005	n.d.	n.d.	0.002	n.d.	n.d.	0.024	0.026	n.d.	n.d.	0.445	n.d.	99.59
C-P1/13	0.002	n.d.	0.001	n.d.	0.004	n.d.	0.019	0.001	0.003	n.d.	0.087	0.031	100.17
C-P1/13	0.002	n.d.	n.d.	n.d.	0.004	n.d.	0.024	n.d.	0.015	n.d.	0.254	n.d.	99.57
C-P1/13	n.d.	n.d.	0.007	n.d.	0.013	n.d.	0.011	n.d.	0.024	n.d.	0.066	n.d.	99.57
C-P1/13	0.002	n.d.	n.d.	0.004	n.d.	n.d.	0.010	0.016	0.014	n.d.	0.211	0.010	99.91
C-P1/13	n.d.	n.d.	0.006	n.d.	0.014	n.d.	0.029	0.026	0.007	n.d.	0.343	0.017	100.03
C-P1/13	n.d.	0.002	n.d.	0.003	0.009	0.034	0.014	n.d.	0.012	n.d.	0.469	n.d.	99.68
C-P2/13	0.005	n.d.	n.d.	0.002	n.d.	0.016	0.002	0.025	0.037	n.d.	n.d.	0.014	99.99
C-P2/13	0.005	n.d.	n.d.	0.001	n.d.	n.d.	n.d.	0.006	n.d.	n.d.	0.007	n.d.	99.41
C-P2/13	0.005	0.015	0.005	0.009	0.006	0.011	n.d.	0.013	0.026	n.d.	0.016	n.d.	99.76
C-P2/13	0.006	n.d.	0.003	0.002	0.001	n.d.	0.002	0.023	0.007	n.d.	0.014	n.d.	99.52
C-P2/13	0.005	n.d.	0.003	0.003	0.007	0.020	0.002	n.d.	0.015	n.d.	0.003	n.d.	99.73
C-P2/13	0.004	0.007	0.005	n.d.	0.015	n.d.	n.d.	n.d.	n.d.	n.d.	0.008	0.019	99.68
C-P2/13	0.004	0.009	0.005	0.002	0.014	n.d.	0.007	0.004	n.d.	n.d.	0.034	0.002	99.65
C-P2/13	0.007	n.d.	0.001	0.009	n.d.	n.d.	n.d.	0.005	n.d.	n.d.	n.d.	n.d.	99.82
C-P2/13	0.005	0.004	n.d.	0.011	0.008	n.d.	n.d.	0.018	n.d.	n.d.	n.d.	n.d.	99.79
C-P2/13	0.001	0.004	0.002	0.004	n.d.	0.003	n.d.	0.011	n.d.	n.d.	n.d.	n.d.	99.90

Table 6a. LA-ICP-MS analysis of copper metal phases in production debris and artefacts. Values sought but not found above the indicated detection limit were treated as not detected (n.d.).

	Cu	Co	Ni	Zn	As	Ag	Sb	Te	Pb	Bi
	%	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
detection limit		*1*	*20*	*5*	*37*	*4*	*5*	*2*	*2*	*1*
P10/13	100.0	23	84	31	bdl	7	bdl	bdl	bdl	bdl
C_P2/13	100.0	41	21	27	bdl	6	bdl	bdl	bdl	bdl
C_P1/13	98.2	bdl	82	131	bdl	230	19	bdl	17800	130

picture of the evolution of metallurgy in the Balkans, from the core Vinča sites to the entire region, and beyond.

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Appendix

Appendix avaliable online as part of Appendix B at https://doi.org/10.32028/9781803270425/AppendixB_Ch26

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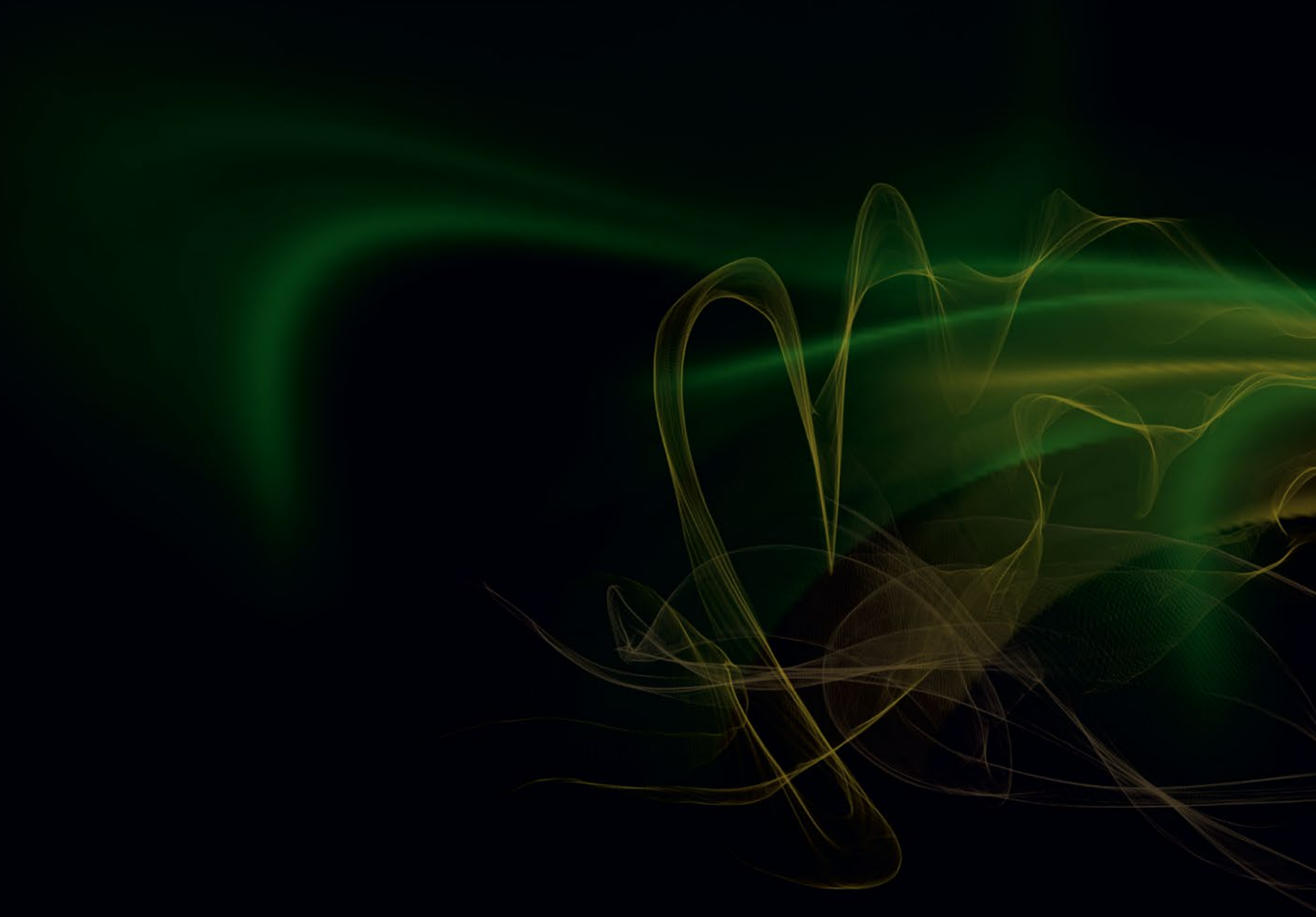

The Rise of Metallurgy in Eurasia is a landmark study in the origins of metallurgy. The project aimed to trace the invention and innovation of metallurgy in the Balkans. It combined targeted excavations and surveys with extensive scientific analyses at two Neolithic-Chalcolithic copper production and consumption sites, Belovode and Pločnik, in Serbia. At Belovode, the project revealed chronologically and contextually secure evidence for copper smelting in the 49th century BC. This confirms the earlier interpretation of c. 7000-year-old metallurgy at the site, making it the earliest record of fully developed metallurgical activity in the world. However, far from being a rare and elite practice, metallurgy at both Belovode and Pločnik is demonstrated to have been a common and communal craft activity.

This monograph reviews the pre-existing scholarship on early metallurgy in the Balkans. It subsequently presents detailed results from the excavations, surveys and scientific analyses conducted at Belovode and Pločnik. These are followed by new and up-to-date regional syntheses by leading specialists on the Neolithic-Chalcolithic material culture, technologies, settlement and subsistence practices in the Central Balkans. Finally, the monograph places the project results in the context of major debates surrounding early metallurgy in Eurasia before proposing a new agenda for global early metallurgy studies.

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